EXPANSION OF TOBACCO WITH CARBON DIOXIDE PM 620

Dr. Roger Z. de la Burde and Mr. Patrick E. Aument of the Research and Development Department of Philip Morris, Inc., have disclosed to me the results of their investigation into the expansion of tobacco with carbon dioxide.

The process of the invention concerns primarily the impregnation of tobacco filler with carbon dioxide. The filler is immersed in liquid CO_2 at a temperature not less than -2°C, the liquid is drained away, and the gas is vented to bring the pressure to atmospheric with the result that solid CO_2 is formed on and in the filler. This is then subjected to heating by known methods to bring about puffing of the filler. There is little or no loss of moisture and excellent retention of water extractables.

The desirability of expanding tobacco to give filler of lower bulk density and greater filling power is well known. A great many agents have been proposed to accomplish this. Some of these may leave residues which, though small, are foreign to tobacco, and thus presumably undesirable. Others are naturally present in tobacco but the residues might, in certain circumstances, exceed the naturally occurring levels. Water is no doubt the least expensive and least questionable additive from a residue standpoint, but means for achieving high levels of puffing require special and costly steps for impregnation. Most of these agents also remove a portion of the extractable components from tobacco during or prior to the expansion.

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Carbon dioxide, like water, would leave a residue (if any) entirely unnoticed by the smoker. It also would be harmless to handle during processing, as well as non-flammable. In Hawkins, U.S. 1,789,435, carbon dioxide is disclosed as one of the gases for impregnating tobacco to produce expansion by abrupt pressure release. However, the degree of expansion realizable from gas impregnation and puffing is relatively small. Efforts to impregnate tobacco with liquid carbon dioxide for subsequent puffing had been unsuccessful. Only by causing CO_2 gas to react with ammonia within the tobacco and by subsequent heating was good expansion realized (Armstrong et al, U.S. Appl. Ser. No. 68,532).

The present inventors have found that it is possible to impregnate filler with liquid CO_2 by maintaining the temperature at -2°C or higher (at the corresponding equilibrium pressure as a minimum), apparently thus avoiding freezing of the moisture in the cells which may exclude the impregnant. The moisture in tobacco absorbs enough soluble matter from tobacco to lower its freezing point; in addition, the CO_2 would cause further lowering. After a soaking period of 5 to 20 minutes, the excess liquid is drained, and the pressure is relieved by venting to bring to atmospheric pressure.

The CO_2-impregnated filler then is heated, as for example in a turbulent air stream at 38° to 66°C, to bring about expansion. Other known heating means may be used, as for example infra-red heating on a belt, exposure in a cyclone dryer, or superheated steam in a tower (or a mixed atmosphere of steam and other gas). The maximum temperature applied will be determined by the length of exposure, CO_2 absorbed, and moisture content among other considerations, but will probably not be higher than 232°C, while the preferred range is 38 to 66°C.

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The tobacco leaf to be expanded may be in uncut form or in large pieces, but preferably is in shreds as used in filler so that no further cutting will be necessary. A moisture content of 8 to 18% (total weight basis) before impregnation is preferred; however, it is believed the method can be used with moisture levels near zero to about 50%. Moistures as stated herein are determined as oven-volatiles (O.V.) by the standard method, but not more than about 2% (absolute) of this value is other than water.

The impregnation takes place in equipment designed to withstand the necessary pressures of 900-1000 psig. The arrangement is not important so long as there is a valved inlet from a source of liquid CO_2 and one valved outlet at the bottom of the vessel whereby liquid may be drained off; a second valved outlet near the top, for venting, may be added, and it might be inserted as part of the inlet line if desired, placed between the vessel and the inlet valve. A means of heating the vessel, such as external coils, is necessary. Supporting the vessel on a load cell greatly simplifies measuring the CO_2 charge. A supplementary vessel similarly equipped with weighing means and heating coils is advantageous, though not essential, because it permits preheating a charge of liquid CO_2 from its usual low storage temperature (at say 300 psig) to perhaps 24°C (900 psig). This helps prevent solid CO_2 formation on charging the main vessel, and shortens the time the tobacco may be below the freezing point of its moisture. The filler is placed in the main vessel in a suitable holder such as a wire basket. The closed vessel is purged with CO_2 gas and the outlets closed, then liquid CO_2 is introduced at 250 psig sufficient to cover the contents. The temperature is raised without delay by the heating coils to at least -2°C but less than 31°C (the critical temperature) and this condition is maintained for 5 to 20 minutes while impregnation takes place. The liquid is then drained off by opening the lower outlet to a suitable reservoir or the like disposal system, and when all liquid is removed the vessel is vented to atmospheric pressure.

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The tobacco, which now contains about 8 to 20% of CO_2 by weight, most of it presumably solid under these conditions, is removed and passed through any of several rapid heating systems to achieve puffing. Systems for this expansion process are most satisfactory which provide rapid, turbulent contact with the hot gas or vapor. With proper control of temperature and exposure time the product may be recovered in the puffed state at a desired moisture content such as 12% so that no reordering is required as it is with product from many expansion processes.

The attached memo of April 20, 1973, inventors to Mr. Burns, shows a schematic diagram of a possible arrangement of equipment to carry out the impregnation process of the invention (Figure 1), together with operating instructions. Figure 2 is a revised and improved form of Figure 1. The reheating of the vessel in Step 14 is now considered unnecessary; it appears in Example III. "Liquidor" is a (registered?) trade name of Air Reduction Co., Murray Hill, N.J., 07974.

The invention is illustrated by the following examples.

EXAMPLE 1

One pound of bright filler at 12% moisture was enclosed in a wire cage and placed in a pressure vessel. After purging with CO_2 gas, CO_2 liquid was introduced to a sufficient level to cover the tobacco sample. The pressure was raised to 600 psig by heating the vessel and was held for 20 minutes. After the equilibration period the pressure was reduced to 350 psig and subsequently held for an additional 5 minutes. The excess CO_2 liquid was drained and gas vented rapidly so as to form solid CO_2 in the tobacco structure. Two samples of the filler were taken. The first sample was spread in a thin layer and allowed to come to room temperature (21°C). The second was gently heated by a hot air flow (about 66°C). This heating gasifies the CO_2 which exerts an expansionary pressure.

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The results are listed below:

Sample	Filling Power, cc/10 grams	% O.V.
Control	33	13.2
21°C	53	13.5
66°C	62	12.2

The data show that the expansion due to the vaporization of CO_2 solid was considerable and that the negligible loss in O.V. eliminated the reordering step, used currently in other expansion techniques. Other heating methods (quartz lamps, heated belts) also were effective in expanding filler impregnated in this way.

The pressure reduction to 350 psig was experimental and probably did not contribute to the results.

EXAMPLE II

Five pounds of bright filler at 12% moisture was impregnated with CO_2 liquid as described in Example I. The sample was expanded by a hot air flow (66°C). Samples of the unexpanded filler (control) and CO_2-expanded filler were submitted for analysis of the major chemical constituents. The results of those analyses are listed below:

Analysis	Control	CO_2 expanded
Moisture (O.V.)	11.8-12.5%	12.5%
Total Alkaloids	2.1-2.3%	2.1%
Reducing Sugars	9.8-12.1%	11.8%
Glycerine	1.5-1.72%	1.62%
Propylene glycol	1.5-1.7%	1.5%

The CO_2 expanded filler shows little change from the control, and this preservation of original components is important.

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EXAMPLE III

The equipment shown in schematic Figure 2 was employed. "Liquidor" I was loaded with 150 lb. of liquid CO_2 from the supply at 250 psig and shut off, then heated to 24°C (900 psig). Two pounds of MF bright tobacco with nominal 12% O.V. was placed in the basket in "Liquidor" II which was then sealed. "Liquidor" II was flushed with CO_2 gas, closed, and 50 lb. of liquid CO_2 was introduced (this was known to be sufficient to cover the basket). The second "Liquidor" was then closed off and heated to 24°C (900 psig). After 10 minutes the liquid CO_2 was drained off by opening the lower valve; this required about 30 sec. The vessel was closed and reheated to 8°C (about 600 psig), then vented at the top to atmospheric pressure in about 30 sec. When the filler was subjected to heating it expanded as did that in preceding examples.

EXAMPLE IV

One pound of 12% O.V. bright cut filler was impregnated with CO_2 gas at a pressure of 900 psig. After an equilibration time of 10 minutes the gas was vented to the atmosphere and the sample was removed. A portion of the filler was heated at 66°C in a low velocity air stream. A second portion was allowed to come to ambient temperature conditions (21°C). The results are listed below:

	Temperature	Filling Power, cc/10 g
Sample 1	66°C	37
Sample 2	21°C	34
Control	-	32

The results indicate that the immersion of the filler in the carbon dioxide liquid seems necessary and that the entrapment of CO_2 under pressure followed by the pressure release does not exert sufficient force to effect expansion.

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The results of any further development of the expansion of tobacco with carbon dioxide will be reported and will become a part of this disclosure.

G. Esler Inskeep

G. Esler Inskeep
Assistant Patent Officer

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